

ISOPROPYL ALCOHOL REFINING

NOVA CHEMICALS LTD.

MOORE SITE

FINAL REPORT

INDUSTRIAL WASTE DIVERSION PROGRAM

JULY 1997



Ontario

**Ministry of
Environment
and Energy**

ISBN 0-7778-6503-3

ISOPROPYL ALCOHOL REFINING
NOVA CHEMICALS LTD.
MOORE SITE
FINAL REPORT
INDUSTRIAL WASTE DIVERSION PROGRAM

JULY 1997



Cette publication technique
n'est disponible qu'en anglais.

Copyright: Queen's Printer for Ontario, 1997
This publication may be reproduced for non-commercial purposes
with appropriate attribution.

PIBS 3564E

ISOPROPYL ALCOHOL REFINING
NOVA CHEMICALS LTD.
MOORE SITE
FINAL REPORT
INDUSTRIAL WASTE DIVERSION PROGRAM

Report prepared by:

Ross Moreton
LDPE Area Work Team
February 1997

Report submitted to:

Waste Reduction Branch
Ontario Ministry of Environment and Energy
under terms of an
Industrial Waste Diversion Program Agreement
dated August 10, 1994

DISCLAIMER

This report was prepared for the Ontario Ministry of Environment and Energy as part of a Ministry-funded project. The views and ideas expressed in this report are those of the authors and do not necessarily reflect the views and policies of the Ministry of Environment and Energy, nor does mention of trade names or commercial products constitute endorsement or recommendation for use.

TABLE OF CONTENTS

1.0	Abstract	1
2.0	Introduction	2
2.1	Project Objective	2
2.2	Project Description	2
2.3	Project Justification	3
2.4	Rejected Alternatives	4
2.5	Outline of MOEE Proposal	5
3.0	The Project	7
3.1	Process Design and Detailed Engineering	7
3.2	Installation	7
3.3	Commissioning	7
3.4	Problems Encountered	8
4.0	Results and Conclusions	12
4.1	Process Modifications Completed	12
4.2	Pending Process Modifications	13
4.3	Objectives versus Results	13
4.4	Recommendations for Others	14

Attachments

1	Process Design Outline
2	Process Flow Diagram
3	Process and Control Drawing

1.0 ABSTRACT

The Low Density Polyethylene (LDPE) process at Nova Chemicals uses isopropyl alcohol (IPA) as a chain transfer agent. A purge stream is removed from the process to control contaminant buildup by distillation. Off the bottom of the distillation column a spent IPA stream is taken which contains IPA with some light and heavy polyethylene oils and waxes.

This spent IPA stream was stored and shipped to a rerefiner for recovery of the IPA. This firm used a batch distillation operation with an oversized distillation column, which recovered approximately 65% of the IPA contained in the feed stream. This recovery level was low.

In this project Nova Chemicals installed facilities at the Moore manufacturing site, near Samia, to distill the spent IPA onsite. The main piece of new equipment was a distillation tower custom designed to maximize the recovery of IPA. Equipment associated with the tower was a reboiler, a condenser, a product cooler, a bottoms pump, and process controls and instrumentation.

Nova Chemicals also installed offsite facilities to provide access to both onspec and offspec tankage for the streams exiting the new distillation tower, to provide utility streams to operate the tower, and to provide electrical power and control for the new facilities.

The MOEE contributed \$228,735 as part of the Industrial Waste Diversion Program to encourage development and implementation of this project.

The project had three main justification elements:

Environmental: Due to the superior fractionation with a tower custom designed for this separation, the amount of unrecovered IPA being incinerated was projected to drop by 300 tonnes/yr.

Transportation: The minimization of dangerous goods being transported over the public highways would improve public safety.

Economics: This project would reduce the amount of fresh IPA purchases required and so would reduce the cost of operation of the site.

At the time of writing this final report the facilities were not yet fully functional, although much progress had been made through various commissioning activities which are described in the body of this report. The main issues have been troubleshooting the onstream analyzer and reduction of swings in the feed flow to the IPA distillation tower.

It is expected that the facilities will become fully functional in the near future. Several periods of operation have demonstrated product purity levels in the 95% purity level, which is the project target.

2.0 INTRODUCTION

2.1 Project Objective

The objective of this project was to refine the LDPE spent isopropyl alcohol (IPA) stream onsite, which would eliminate offsite shipping for IPA recovery. This would result in reduced impact on the environment, improved public safety, and annual cost savings.

2.2 Process Description

Spent IPA Stream

The LDPE process uses IPA as a chain transfer agent. A small portion of the IPA is lost with the product and some is removed in the process recycle entrainment separators. However, most of the IPA remains with the recycle ethylene. A purge stream, taken off the recycle ethylene, is fractionated in the purge column to remove contaminants that build up in the process.

Automatic releases to the flare system from the purge column control the concentrations of light material that builds up in the process. Heavy material is taken off the bottom of the fractionator and contains essentially all the IPA which enters the purge column as well as light and heavy polyethylene oils and waxes.

This bottoms stream is depressured into the oil tank, where lighter material is flashed off and recovered. The bottoms off the oil tank constitutes the spent IPA stream. This material is sent to a rerrefiner for IPA recovery. The spent IPA stream contains all the material generated by the LDPE reaction process that boils between ethylene and approximately a C20 carbon number.

Through a 1994 spent IPA testing program with Shell Chemicals at their Oakville Research Center, it was learned that the spent IPA is made up of over 100 components that form a boiling continuum. The IPA elutes as a large spike that boils slightly lighter than octene. The nonideality of the mixture and the presence of this wide range of components makes recovery of the IPA at high purity levels somewhat difficult.

Rerrefiner Operation

The rerrefiner recovers solvents for many companies. To distill the spent IPA they employ a batch operation using a 64 tray, 48" diameter bubble cap column. The procedure is labour intensive and arduous, involving several hours of total refluxing and cutting material to several different tanks as the distillation process progresses.

There is a cost incentive to recover as much of the IPA as possible and the rerrefiner is using the facilities they have to the best advantage to do this. There is no way they can make an improvement to their operation with the existing facilities.

The rerrefining process loses approximately 1/3 of the IPA in the feed stream to waste. Fresh IPA must be purchased from Shell to replace these losses.

2.3 Project Justification

ENVIRONMENTAL

Due to the superior fractionation with a tower custom designed for this separation, the amount of IPA being disposed will drop by 300 t/yr. This is a 43% reduction in waste generation and in impact on the environment from this source.

TRANSPORTATION RISK

The minimization of dangerous goods being transported over the public roadways will improve public safety in line with Responsible Care policies. Responsible Care is a set of initiatives undertaken by the Canadian Chemical Producers Association (CCPA) to help safeguard employees, the environment, and the communities with which the industry comes into touch. At the current rate of trucking accidents, a truck carrying Moore Plant IPA can be expected to be in an accident once every 100 years.

ECONOMICS

1) Savings versus rerefiner operation

Based on actual numbers from January 1990 through August, 1993 the cost breakdown for the original operation was as follows:

data:	IPA to rerefiner:	853 t/yr @ 83% IPA	
	IPA from rerefiner:	493 t/yr @ 97.5%	
	IPA recovery:	$(493 \cdot .975) / (853 \cdot .83) = 67\%$	
	fresh IPA usage:	473 t/yr	
costs:	fresh:	$473 \text{ t/yr} \cdot \$.70/\text{kg} =$	331 k\$/yr
	recycled:	$493 \text{ t/yr} \cdot \$.32/\text{kg} =$	148 k\$/yr
	IPA to disposal:	$227 \text{ t/yr} \cdot \$.15/\text{kg} =$	34 k\$/yr
	oil/waxes to disposal:	$133 \text{ t/yr} \cdot \$.15/\text{kg} =$	20 k\$/yr
	total:		533 k\$/yr

For the onsite refining facilities the cost breakdown was:

data:	IPA to tower:	853 t/yr @ 83 % IPA	
	IPA from tower:	686 t/yr @ 98% IPA	
	IPA recovery:	$(686 \cdot .98) / (853 \cdot .83) = 95\%$	
	fresh IPA usage:	282 t/yr	
costs:	fresh:	$282 \text{ t/yr} \cdot \$.70/\text{kg} =$	197 k\$/yr
	IPA to disposal:	$36 \text{ t/yr} \cdot \$.14/\text{kg} =$	5 k\$/yr
	oil/wax to disposal:	$133 \text{ t/yr} \cdot \$.14/\text{kg} =$	19 k\$/yr
	tower operating costs:		20 k\$/yr
	total:		241 k\$/yr

Net savings = 292 k\$/yr. The Fall 1994 expansion increases this by 20% to 350 k\$/yr

Net IPA recovery = 191 t/yr. The Fall 1994 expansion increases this by 20% to 230 t/yr

2) IPA recovery from Oil Tank Overhead

The current operation runs with the oil tank at about 20 psig and 50 deg C to flash off volatiles before the spent IPA reaches the spent IPA tanks, which operate at atmospheric pressure. In

so doing some IPA is lost to the flare system. Addition of the IPA refining tower will allow the oil tank to be run at a higher pressure resulting in a reduction in IPA losses to flare by 70 t/yr, worth 49 k\$/yr, after the Fall 1994 expansion.

3) Other Savings

Increased propylene Recovery:	9	k\$/yr
Reduced trucking costs:	34	k\$/yr
Reduced use of lugger buckets:	12	k\$/yr

The total annual savings generated by this project are therefore 454 k\$/yr.

2.4 Rejected Alternatives

Several alternatives were examined to determine the best process solution

1. Have Shell Rerefine the Spent IPA

Shell was approached regarding the potential for them to reprocess our Spent IPA. Shell make a pharmaceutical grade of IPA at 99.8% purity. Their IPA production process generates five specific contaminants which they remove using four separate distillation columns because of the non-ideal and azeotropic nature of the distillations. The only significant contaminants in the finished IPA product are normal propanol and water. All other contaminants are reduced to PPM levels.

In comparison, our Spent IPA has over 100 contaminants, many of which cannot be precisely identified. Shell were extremely reluctant to enter into any processing situation whereby they might contaminate their high purity product. This possibility was not pursued further.

2. Inject the Spent IPA as a Gasoline Additive

Because of the small spent IPA volumes, it is probably feasible to flash off the IPA from the spent IPA stream and blend this material into gasoline. The IPA would act as an oxygenate and would improve the combustibility of the gasoline. The existing facilities at Moore would require some modifications to flash and condense the material, and an unloading system, storage system, and injection system would be required at the Corunna site. A ballpark "guesstimate" of capital costs at the two sites would be in the 250 to 500 k\$ range.

However, the value of IPA as gasoline is only 40% of the IPA value, so the incentives are not favourable. The main difficulty is that, with no IPA being recovered, all the IPA leaving the process will end up in gasoline and therefore all the IPA used at Moore will be full cost fresh IPA from Shell. So the credits of spent IPA to gasoline are offset by the increased cost of the IPA required at Moore and the net benefit is only 35 k\$/yr. This was not a worthwhile option.

3. Burn all the Spent IPA to capture fuel value

This option is similar to the gasoline injection option above, except we simply burn the spent IPA stream in the boilers to recover the fuel value of the material. The capital cost would be in the 200 to 300 k\$ range, but, because fuel value is even lower than gasoline value, this option would actually cost us 50 k\$/yr more than the current operation.

4. Sell all the Spent IPA to other industry

Finding a firm interested in our Spent IPA to use as a feed to their process would have been a good alternative. Facility changes would be minimal versus the current operation.

As part of the development activities for this project, several companies were contacted through the Ontario Waste Exchange regarding potential sale of the spent IPA. There was minimal interest. Two remarketers did some analyses of the spent IPA to determine what might be the best use for this material, and whether there might be some pretreatment that could be effective in making it more salable. This work did not turn up anything useful.

5. Modify Purge Column to fractionate out the IPA

The existing purge column was simulated to determine whether any opportunity exists to improve the fractionation and so minimize the facilities that might be required to fractionate the IPA out of the spent material.

It is not possible to simply fractionate out the IPA in the purge column, however by increasing the bottoms tray temperature through increased reboiler duty some of the lighter components can be removed from the purge column bottoms stream. Unfortunately, a large change in the bottoms temperature is needed to produce any significant modification, which changes the composition of the side draw to flare considerably (heavies it up), and forces ethane up the tower.

These changes are not felt to be acceptable for maintaining good purge tower operation. Furthermore, in spite of the decrease in light materials in the purge column bottoms, the purity is still not sufficiently high and a fractionation tower is still required.

6. Use a simple flash and condense approach

Instead of a distillation column, simple flash off the IPA and lighter from the heavy oils and waxes, then condense out the IPA from the vapour stream letting the uncondensed light material go to the flare. Simulations of this type of operation show that a purity of 90% by weight is probably the best that can be achieved with an 80% IPA recovery.

This purity level is not sufficient for use of the material as catalyst carrier IPA and reactions would occur before reaching the reactor. Therefore this approach is not viable.

2.5 Outline of MOEE Proposal

The proposal to the Ministry of the Environment and Energy was for facilities to be installed at the Nova Chemicals Moore Site to refine the spent IPA. The main piece of equipment would be a new fractionation column. Required new facilities directly associated with the tower would include a reboiler, condenser, product cooler, cooling water supply, steam supply, and instrumentation.

It was proposed that this unit be constructed as a package facility, meaning all components associated with the tower would be assembled in the vendor shop.

Maximum use would be made of the existing process facilities. The existing oil tank would be operated at a higher pressure to avoid the need for a pump and to recover the IPA currently being discharged from the oil tank overhead to the flare. The bottoms from the tower will be directed to the existing waste tank for disposal to Laidlaw. One of the existing spent IPA tanks

will be used for the IPA product to verify composition before sending it to the existing IPA surge tank.

The unit would be located on the existing curbed concrete pad just to the north of the existing oil tank. Process and utility tie-ins were readily available at this site. A scoping type estimate (+/-30%) was prepared for this project. The breakdown was as follows:

<u>Equipment</u>	<u>k\$</u>	<u>k\$</u>
Tower	78	
Exchangers	39	
Piping	139	
Pumps	13	
Instrumentation	102	
Electrical	35	
Concrete	10	
Structural	9	
Equipment Rentals	10	
total direct material and labour		435
Delivery	25	
Construction mgmt/sup'n (8%)	35	
Engineering (12%)	52	
total indirects		112
contingency (25%)		<u>128</u>
grand total		675

3.0 THE PROJECT

3.1 Process Design and Detailed Engineering

In the expectation of minimizing capital costs for this project, bids were solicited from several vendors of package units. They would handle the process design, detailed engineering, and construction for the main distillation and ancillary equipment.

Glitsch Package Division was the selected vendor. Attachment 1 contains the technical package sent to each of the possible vendors for them to provide a bid package for the facilities. Attachment 2 shows a simplified process flow diagram and Attachment 3 shows a detailed Process and Control Diagram.

The rest of the facility design and connection of the package unit into existing facilities was handled onsite by Nova personnel. Glitsch delivery was 4 months late. They had promised delivery by the end of August 1994, however actual delivery was mid-December of that year.

3.2 Installation

Preparatory work for installation of the package unit had been proceeding during 1994 and the various utility system and process tie-ins were available well ahead of the actual delivery date. The unit was removed from the flatbed truck and hoisted into place and bolted down shortly after arrival.

During the January to March time period in 1995 the final construction activities and checkouts took place and the unit was available for commissioning work by April 1995.

3.3 Commissioning

Plans were made to carry out the initial commissioning for the IPA Refining Project, including:

- 1) Ensure that pre-commissioning was complete
 - analyzer
 - tags
 - computer control
 - alarm points
 - safety reviews
 - procedure changes
- 2) Set up piping to direct both make IPA and bottoms to D-538. Essentially this would split the stream in the IPA column and put it back together downstream. The light vapours would be directed to flare. After the initial testing was done a decision would be made when to swing the product IPA to the fresh IPA surge tank.

- 3) Unit integrity and operation tests
 - use spent IPA to check out actual unit operation using the detailed procedures provided by the package unit vendor
- 4) Unit optimization tests (most of these have not yet been done)
 - min/max operating rates
 - feed nozzle location
 - product nozzle location
 - setting for tray 40 liquid purge
 - setting for tray 35 liquid purge
 - performance guarantee check
 - optimal purity setting (recovery vs purity)
 - best control strategy

3.4 Problems Encountered

It was expected that there would be some difficulties to overcome to optimize this process due to the uncertainties surrounding the feed composition and the fact that for different reactor grades the operating conditions and amounts of various resin modifiers, including IPA, is substantially different.

In reality, the commissioning work on this project has been much more onerous than anticipated, with several modifications being made to the IPA Refining unit itself and to upstream equipment. The key issues and actions taken are summarized as follows, in rough chronological order:

- 1) Analyzer Sample Tap: There was an initial problem getting a flow to the analyzer. There appeared to be too much vapour in the top of the line resulting in vapour recirculation back to the product line from the sample return stream. This problem was resolved by rotating the section of line to allow the sample to be taken off the side of the line.
- 2) Feed Flow Meter Flashing: After startup erratic readings were noticed from the tower feed flow meter. This was resulting from flashing due to excessive pressure drop across the feed control valve. Piping modifications were made to move the flow orifice upstream of the feed control valve, which resolved the problem.
- 3) Bottoms Thermal Relief Valve Lifting: Early on it was noticed that the bottoms stream thermal relief valve was passing by. This is probably the result of the valve not reseating correctly due to the waxy nature of the material. We have had a recurrence of this problem recently and have steam traced the thermal relief valve and the inlet and outlet piping to prevent the heavy material from solidifying.
- 4) Analyzer Suction Vapour Trapping: Due to continued problems getting a feed flow to the analyzer a vent line was installed to facilitate removal of vapour from the purified IPA line. This change appeared to resolve this issue.
- 5) Oversized Bottoms Pump: The bottoms pump supplied as part of the package unit was significantly oversized by a factor of 10 times. We followed up with the package unit and pump vendors and decided to fit the pump with reduced trim. The first version of the new trim came with incorrect gear material and was damaged quickly. This was traced back to a swelling problem and the correct version of the gears was purchased. This problem was ultimately corrected.

- 6) **Analyzer Problems:** A major problem occurred with the online analyzer which took several months to sort out. Initially, there was significant plugging with wax which we attributed to the unsuccessful commissioning activities we had been experiencing to date. This was resolved with a change to the control strategy on the tower to avoid high temperature situations in the bottom of the tower which resulted in wax being vapourized and carried out with the purified IPA.

However, we continued to experience continually dropping purity readings, as verified with outside lab testing. This led to a major investigation of the analyzer operation involving discussions with the analyzer manufacturer and a site visit. Many modifications and testing were done to determine the cause of the problems, which included installing filters, replacing tubing, replacing seals, replacing shafts, changing standard mixtures, changing analyzer cycle times, and changing analyzer operating temperature.

Ultimately, after numerous meetings, equipment changes, and testing we determined that the sample valve seals were made from an inappropriate ceramic material which broke down and caused the formation of a black gritty material as the valve stroked. This material plugged up the portion of the valve that was intended to deliver the sample to the instrument. It was ultimately decided to completely replace the analyzer sample valve with a different design. This resolved the seal erosion problem.

The other significant issue which was uncovered was density differences, due to temperature changes in the sample conditioning system, were causing significant deviations in the analyzer feed flow and therefore product purity readings. These fluctuations were resolved by installing a preheating loop in the analyzer heating chamber to ensure the sample temperature was consistent regardless of the analyzer feed flow.

- 7) **IPA Product Condenser Oversized:** The product condenser supplied with the package unit was significantly oversized. This, combined with the fairly wide variability in the purified product flow, created a situation at the lower feed flow rates where excessive condensation of vapour was taking place. The excess condensate was returning to the main distillation tower by gravity.

The concern with excess condensation is that the purity of the purified IPA product stream will decrease. The objective is to only take out of the column that material which will be the purified IPA and condense it totally in the condenser.

The initial fix was to rework the piping to prevent condensed material draining back to the distillation tower. This forced all condensate to remain in the condenser, resulting in a level increase in the condenser.

The excess condensation problem was alleviated. However, during the ensuing Winter, hammering started in the condenser. Apparently, the vapour space in the condenser had totally disappeared and the vapour entering the condenser was no longer being condensed by the cold tubes of the tube bundle.

The tubes were cooling the condensate to low levels and the condensate itself was cooling the incoming vapour. This caused rapid collapse of the vapour bubbles and hammering in the condenser. To overcome this some steam tracing was wrapped around the top foot of the condenser to prevent the liquid level from reaching the top.

- 8) Oil Tank Flow Erratic: It was noted after startup that the flow from the upstream surge drum was erratic and varied in a range of about 10% from the average. The control valve in the system was operating close to the closed position, meaning that small movements of the valve stem would cause large relative changes in the position and therefore the flowrate through the valve. After living with this for a while, it was decided to install a reduced trim in the valve to maintain a steadier flow rate.
- 9) Bottoms Check Valve Passing: The initial design specified a check valve in the bottoms line with a spring device to maintain a certain back pressure on the system. The intent of the spring was to prevent the tower contents from draining out of the unit through the positive displacement pump when the pump was not running or when the tower itself was shutdown.

It was discovered that the seat in the check valve was sealed with a plastic material which was eroded by the flow through the valve. After several replacements, thinking that this was a startup grit problem, a control valve was installed to prevent unwanted loss of the tower bottoms material. This has worked well.

- 10) Upstream Operation Erratic: The tower feed flow underwent extreme fluctuations, more than the surge capacity in the oil tank could smooth out. This was traced back to control of the purge column upstream of the IPA Refining column. Some tuning of the upstream column control loops was carried out, however the problem was not easily fixed.

By the end of 1995, after 8 months of commissioning activity, it was decided that a significant effort would be needed to modify the operation of the purge column. A team was formed and several meetings held. Many changes were made to the purge column operation, the most significant of them being:

- increased swinging of upstream exchangers to minimize feed vapourization and flow variability
- carried out a capital project to install an additional control valve to control the side feed temperature
- reworked the tower control strategy maintain a constant ratio between the top and side feed rates
- reduced the area of the reboiler, which was significantly oversized
- changed the reactor control scheme to avoid use of purge column for reactor concentration control

Not all aspects of the above changes are completely finished, however we were able to restart commissioning work in November of 1996, with more success.

- 11) Top Tray Flow Control: One of the features of the IPA Tower control strategy is the ability to remove a liquid stream from both tray 35 and tray 40. These had been arranged as manual devices using rotameters and ball valves based on the expectation that these flows would be small. They tied together with the tower bottoms flow upstream of the bottoms pump.

Our current understanding is that these flows are more significant than originally thought and may be the most significant in terms of maintaining product purity. The tower overhead

vapour flow to the flare has been minimal and the bottoms flow has been somewhat less than anticipated.

We have found that when the bottoms level control circuit goes to zero flow to control a decreasing level, there is no outlet for material that is building up on the upper trays in the tower. It is too heavy to stay as a vapour and be removed to flare and it is too light to make its way out the bottoms. It starts building up in the tower and eventually is removed with the purified IPA, dropping the purity.

As a stopgap measure we have put a lower limit on the output of the tower level control loop to allow some outlet for this material through the pump. However, this allows minimal control of this stream and is an inefficient operation. We are currently examining installation of a control valve on this stream to allow independent control of this flow.

- 12) Control Strategy Modifications: The original control strategy for the column has been changed a couple of times to allow for smoother operation. The current strategy is straight forward: tower pressure is controlled by the overhead vapour flow to flare, the bottoms temperature is controlled by steam rate to the reboiler, bottoms level is controlled by the bottoms flow, pressure drop across the trays is controlled by the purified IPA flow, and the tray 35/40 liquid flows are manually set.

As of the writing of this report, we have experienced several days of operation with the tower reasonably well lined out and producing onspec purity numbers. We have not been able to, as yet, carry out the normal optimization activities, as documented in the commissioning section above.

4.0 RESULTS AND CONCLUSIONS

4.1 Process Modifications Completed

The following modifications to the original design have been installed on the IPA Refining unit to date:

- 1) Rotated product line to eliminate the possibility of vapour trapping preventing liquid reaching the analyzer.
- 2) Moved the feed flow meter upstream of the surge tank level control valve to prevent flashing of the feed.
- 3) Steam traced and insulated the bottoms pump safety relief valve piping to prevent waxy material from preventing reseating of the valve.
- 4) Installed piping to bring the high point vent to grade at a pipebridge. This allowed easy removal of vapour from the piping.
- 5) Corrected the oversized bottoms pump by installing gearing with reduced width.
- 6) Made several changes to the analyzer to track down a faulty seal problem. We ultimately changed the sample valve completely and installed a sample preheating loop in the analyzer.
- 7) Modified the inlet piping to the product condenser to eliminate the return of condensate back to the tower. Also, some steam tracing was wrapped around the top of the condenser to minimize hammering due to condensation of the incoming vapour by cool liquid in the condenser.
- 8) Installed a control valve on the bottoms stream in series with the bottoms pump to gain positive control of this stream.
- 9) Did a major analysis of the upstream distillation tower operation. This included operational changes, control changes, and installation of additional control hardware.
- 10) Modified the original tower control scheme to allow more direct control of the key tower control parameters, in this case the bottoms temperature and the tower pressure.

The above noted changes have allowed us to achieve a basic tower operation. We are able to maintain reasonable operation by "babysitting" the process and making manual adjustments to the process. There are several further changes that we are planning to make to allow online optimization of the operation. These are described in the next section.

4.2 Pending Process Modifications

The remaining small fixes required to optimize the process and complete commissioning work on this project are:

- 1) Install steam tracing on the overhead vapour to flare to prevent plugging of the pressure control valve due to auto-refrigeration across the valve.
- 2) Install a jumper to allow fresh IPA flow to flush out the IPA product line after purity excursions.
- 3) Install a control valve and flow meter on the tray 35/40 flow to allow monitoring and control of this stream.
- 4) Install a reduced trim in the feed surge drum level control valve to allow smoother control of the feed rate to the IPA column.
- 5) It appears we will have to trace the analyzer feed tubing to prevent plugging during purity excursions.
- 6) Consider installation of a make/vent system to give a more positive control of tower pressure.
- 7) Install reduced trim in the pressure control valve to allow improved control of this variable.
- 8) Bring the top local tower temperature indicators into the control system. We are finding that these are good indicators of the direction the tower performance is moving.

4.3 Objectives versus Results

Waste Reduction versus Plans: Because we have been unable to fully optimize the unit at the time of writing this report, we do not yet have a good analysis of the actual recovery potential. Some preliminary information based on a recent two week period of online operation shows that we are recovering approximately 85% of the incoming IPA. This compares favourably with the 65% recovery by the re-refiner.

We still expect that we will achieve the project target of 90% recovery of the feed IPA after the final hardware changes are installed and optimization of the process is complete.

Actual project Costs versus Plans: A total of 790 k\$ was appropriated to cover the cost of the IPA Refining project. The final cost will be somewhat over 900 k\$ when all the changes are included in the total. The project overrun was mainly due to a significant increase in the above ground piping work required. However, offsetting this somewhat were underruns for the IPA package distillation unit, the civil/structural work, and construction supervision. The reasons for the under and overruns in these categories are:

- Above ground piping overrun: Several changes occurred which led to increases in the above ground piping costs. These included: increase in some line sizes, preparation of tie-in spool pieces, hot taps to the cooling water lines, crane time for piping lifts, scaffolding, replacement of frozen valve, some underground piping done by the piping contractor instead of the civil contractor, and other minor changes/additions.
- IPA distillation unit underrun: The unit was contracted to be paid in US dollars. A change in the exchange rate change caused this underrun.
- Civil/structural work underrun: Some of the underground piping work originally expected to be done by the civil contractor was done by the piping contractor.
- Construction supervision underrun: This was our first time through with the package unit approach, and the actual construction supervision was less than anticipated.
- Commissioning Changes: There have been several changes to the facilities during the commissioning process. Although most of the changes have been small, the cumulative effect has impacted the project cost.

Project Payback versus Plans: The simple payback at the time this project was appropriated, based on the planned 454 k\$/yr savings and a capital investment of 790 k\$ was 1.74. The final capital cost, after all changes are complete, will be approximately 920 k\$. This change in cost will negatively affect the payback, however, there is an offsetting factor.

During the two year commissioning process the price of IPA has escalated by 16%, from \$.70/kg to \$.814/kg. Because most of the project credits are based on IPA cost, the project credits rise to 489 k\$/yr. The resulting simple payback is therefore 1.88, somewhat less favourable than the original number, but still attractive. When the MOEE Industrial Waste Diversion Program grant payment is taken into account the payback becomes even more favourable, at 1.42.

4.4 Recommendations for Others

In light of the difficulty experienced during the commissioning stages of this project, there are several recommendations that can be made that might help direct the efforts of others faced with a similar recovery opportunity.

Feed stream definition: It is of utmost importance to define the feed stream characteristics as well as possible. Nova Chemicals was faced with a difficult situation due to the waxy nature of the feed and were only able to gather one data point through an onerous testing program undertaken by Shell Chemicals on our behalf. We therefore were not able to specify the range and variability of the individual stream components.

Ensure upstream process is under control: We were surprised by the degree of variation in our upstream distillation tower, and the rapidity of the cycling that was occurring. This seriously interrupted the commissioning activities for the IPA Refining unit while we undertook improvements.

Build in Options: As part of the design of the refining facilities alternate feed inlet and product outlet locations were installed to allow optimization of the recovery of IPA. Similarly, light liquid drawoffs were established on trays 35 and 40 to allow alternate locations for removal of light liquid.

Analyzer Location: Our analyzer location is several hundred feet from the distillation tower, which inserts a lag of 1 to 2 hours between the time the material is produced off the tower and receipt by the analyzer. New analyzer equipment is now available which could be located right on the unit. This would reduce the lag time considerably and would be much preferable.

Package Unit Vendor: In retrospect, we feel some additional formal review time during the actual process design stage would probably have been beneficial. This would have added to the project cost but may have helped size the equipment more closely to anticipated flowrates.

Control Scheme: We have discovered that the IPA distillation tower is very sensitive to even small changes in pressure and temperature. The tower control scheme was modified to one that gave direct control and maximum stability to the tower pressure and the bottoms temperature.

Light Liquid Flowrate: The relative amount of light liquid requiring removal from the top trays versus the amount of vapour being discharged to the flare system was a surprise. There was very little vapour and proportionately more light liquid. This is causing us to make some adjustments to the tower equipment to provide automatic control for the light liquid stream.

Purity Control: We have found that the light liquid draw rate is the most important variable to maintain on-spec purity. Due to the amount of material requiring removal from the column from the top trays as liquid, a dedicated control system is needed. Our original process comprised a manual valve with a rotameter to set the flow. This does not give sufficient control for optimization especially when the upstream operation makes several different grades, each with a different IPA injection rate.

Hardware Changes: The completed and pending hardware changes described in Sections 4.1 and 4.2 have been found to be worthwhile adjustments to the original design and should be incorporated in any other similar process.

Attachment 1

Process Design Outline

1.0 GENERAL DESCRIPTION

SCOPE

This project provides onsite distillation facilities to refine the Spent IPA stream. All tie-ins to existing facilities, offsite lines, utilities, and control equipment are included in the project scope.

DESIGN BASIS

UNIT CONSTRUCTION: The new IPA refining column and all associated equipment shall be purchased as a package unit. All detailed process design, detailed engineering, and construction shall be carried out by the package unit vendor. The package unit shall arrive onsite with as much work complete as is practical. Details regarding the breakdown of work expected from the package unit vendor and what is done locally are given in Section 6.

RATE: The process shall be designed to handle a maximum fresh feed rate of 275 kg/hr plus a typical offspec recycle rate of 25 kg/hr for a total rate of 300 kg/hr. The recycle feed rate shall be adjustable. The minimum feed rate shall be 130 kg/hr, with a typical average rate of 180 kg/hr.

PRODUCT PURITY: The IPA product stream shall contain a minimum of 95 wt% IPA.

IPA RECOVERY: The new facilities shall recover a minimum of 90 wt% of the IPA contained in the feed stream. The unit shall be designed for higher recoveries than 90 wt% if feasible and economically worthwhile.

SERVICE FACTOR: The IPA refining facilities shall be designed to require minimal downtime. The overall reaction process is not scheduled for regular annual outages, and shutdowns occur on an "as required" basis for generally short periods of time. The expected service factor for the IPA facilities shall be a minimum of 99%.

PROCESS DESCRIPTION

CURRENT OPERATION: As part of the recycle operation for the LDPE unit, IPA and polyethylene oils and waxes are removed from the process as the bottoms from the purge column. This stream is depressured into the oil tank where evolved vapour recovered. The bottoms from the oil tank constitutes the spent IPA. This material is currently stored in the two spent IPA tanks for shipment to a rerefiner for recovery of the IPA. The purified material is returned to the site into the fresh IPA surge tank, from where it is pumped to the process.

FUTURE OPERATION: This project will make modifications to the current operation to recover the IPA onsite. The operating conditions of the spent IPA tank will be modified to minimize the current loss of IPA to the flare system. The bottoms from the oil tank will be sent to a new distillation column to recover the IPA. The IPA product from the column will be analyzed by an online analyzer and sent directly to the fresh IPA tank while onspec. When offspec, the IPA product stream will be directed to D-538, one of the existing spent IPA tanks.

Light oils and waxes from the new distillation column will be pumped to the gunk storage tank (modifications for this to be provided as part of the Gunk Lugger Replacement Project), and uncondensed light materials will be combined with the overhead from the oil tank.

Provision is made for direction of the spent IPA to the offspec tank during periods of time when the new IPA recovery column may be unavailable or during startups, so that recovery of the contained IPA can be done later. Also, the capability to load trucks with offspec or bypass spent IPA will be retained.

2.0 UTILITIES AND CHEMICALS

200 PSIG STEAM

This steam level will be used to provide heat for the reboiler for the new tower. The exact amount of steam required will not be known until the detailed process design is completed by the package unit vendor. A new 2" line will be needed to bring the steam to the new unit from the piperack.

INSTRUMENT AIR

Additional amounts of instrument air are required to operate the new control valve stations. The required increase in volume of instrument air is available from the existing supply system.

NITROGEN

Small amounts of nitrogen will be required for purging out the unit during shutdowns. A takeoff from the nitrogen supply header will be installed to provide the required amounts.

COOLING WATER

Cooling water is required to condense the tower reflux and to condense the vapour product stream. The exact amounts needed will be firmed up after the detailed process design is completed by the package unit vendor.

CONDENSATE

A connection into the condensate recovery system is available near the new IPA recovery unit. Room exists in the condensate system to handle this additional flow.

DELUGE SYSTEM

The new package unit will be protected from fire by extension of the existing deluge system. This will be carried out by the Vipond Automatic Sprinkler Company, who estimate that between 50 and 100 usgpm of additional flow will be required.

SEWER SYSTEM

There will be a slight increase in the amount of water needing removal during a deluge situation by 3%. The existing sewer system can handle this small increase. All other water sources entering the area will be unchanged, such as fire monitors and rainfall.

75 PSIG STEAM

Steam tracing at the 75 psig level will be required on several of the lines. An existing 75 psig steam header is available adjacent to the package unit location.

3.0 ELECTRICAL

ELECTRICAL CLASSIFICATION

All areas where equipment is to be added or modified as part of this project are classified as Class 1, Group C, Division 2.

RECYCLE PUMP MOTOR

The power requirements for the recycle pump motor will be very small at approximately .01 HP. This energy can most conveniently be supplied at the 120 volt level from a local panel. A local pump stop/start is needed. The motor shall be the TEFC and variable speed with local adjustment to allow flow to be set to the desired rate.

IPA TOWER BOTTOMS PUMP MOTOR

The power needs for the bottoms pump motor will also be quite low, however, because of the need for greater reliability of power supply for this motor, the source of the power shall be at the 575 volt level. The motor shall be the TEFC and variable speed drive to allow flow adjustment to control the level. A local start/stop will be needed.

LIGHTING

Lighting will be needed at the IPA refining package unit. It is expected that a total of four 100 watt, explosion proof, high pressure sodium lights will be required. For estimating purposes it shall be assumed that capacity exists at one of the local panels.

GROUNDING

The new IPA refining unit shall be tied into the grounding circuit in the area.

4.0 CIVIL, CONCRETE, AND STRUCTURAL STEEL

CONCRETE WORK

To support the new package unit four spread footing foundations will be required at the corners of the structure. Parts of the existing concrete pad will be broken out as required to allow this, and replaced. When the concrete is removed the cooling water piping runs under the pad shall be installed. During the detailed engineering phase of this project, when the total weight of the package unit is more clearly known, the need for spread footings shall be reconfirmed versus use of the existing concrete slab.

STRUCTURAL STEEL

All structural steel required by this project shall arrive as part of the package unit. Existing pipe racks are suitable for the new piping required, with sufficient space available.

5.0 BUILDINGS (not required)

6.0 PRESSURE VESSELS AND STORAGE

The IPA Refining Column, complete with all ancillary equipment, will be designed, constructed, and delivered to the site by the package unit vendor. This section describes the various requirements that will govern the design and construction of the package unit.

PACKAGE UNIT SCOPE

INCLUDED: The facilities to be included within the package unit and which will be furnished by the package unit vendor are:

- IPA refining tower complete with all trays and other internals
- Reboiler, bayonet internal type
- Condenser, direct reflux internal type
- IPA product condenser/receiver
- variable speed, positive displacement bottoms pump

- feed strainer sized to capture approximately 1/2" size chunks (during design consider orienting piping to accommodate future filter)
- safety valves for overpressure protection with associated piping
- piping for all process and utility lines to boundary of steel support structure
- all instrumentation for control of the column and associated facilities
- instrument wiring marshalled to a terminal cabinet
- a list of spare parts required for the package unit shall be provided by the vendor

NOT INCLUDED: Facilities which will be needed at the package unit but will not be provided by the package unit vendor are:

- deluge system piping and sprinkler heads
- grounding connection
- foundations
- piping tie-ins
- installation of package unit and final hydrotest
- final calibration of all equipment
- tracing and insulation (after final hydrotest)
- lighting
- analyzer and analyzer tubing

LINES: The specific lines that will be crossing the boundary of the package unit are:

- spent IPA feed
- uncondensed overhead material
- safety valves to flare system
- IPA product
- combined oil and wax from bottoms and top tray liquid draw
- steam in
- condensate out
- cooling water in
- cooling water out
- deluge system in
- nitrogen supply

PACKAGE UNIT PROCESS DESIGN REQUIREMENTS

DUTY: As per design basis in Section 1 of this document

PROCESS DESIGN OPTIMIZATIONS: There are several areas that need optimization or analysis during the detailed process design. They are:

- number of trays
- feed tray location
- product tray location
- reflux rate
- liquid purge rate from top tray
- confirm feed above product is optimal
- tray spacing
- tower diameter
- any impacts due to offspec recycle
- potential for fouling in bottom of tower

PRESSURE REQUIREMENT: Existing facilities will impose some minor restrictions on the design of the package unit. The key one is an allowance for sufficient pressure at the unit to push the discharge streams against liquid levels in tankage. The worst case will be a liquid level of 30 feet in either the IPA surge tank, the offspec tank, or the gunk storage tank. All of the tanks are at atmospheric pressure.

INHERENT SAFETY: The package unit shall be designed with the principles of inherent safety in mind. The key aspects pertinent to this design are: minimization of volumes of flammable fluids contained in equipment, simplicity of operation, and tolerance of poor installation or operation without failure.

CODES AND REGULATIONS: All applicable codes and regulations shall be adhered to. A package of information containing specific Novacor corporate requirements shall be sent to the chosen package unit vendor prior to process design.

METALLURGY

Generally speaking, carbon steel will be an acceptable material for the facilities covered by the package unit. Exceptions to this will be the tubes in the tower overhead and the IPA product condensers, where admiralty brass will be specified to provide superior erosion resistance to the cooling water flow. Also, the valves in the tray decks shall be stainless steel. For the smaller pieces of equipment the standard vendor offerings will normally be acceptable. All of the material selections shall be reviewed with Novacor personnel.

PACKAGE UNIT OPERATING PHILOSOPHY

PREFERENCES: For ease of operation several requirements and preferences are noted here:

- 1) The package unit shall be designed to minimize operator attention.
- 2) All controls shall be from the central control house.
- 3) During upstream process upsets the unit shall be able to go to a total reflux operation to avoid short duration shutdowns.
- 4) Eliminate need for frequent cleaning of facilities. If dewaxing of internals is necessary use fresh IPA and total reflux for a period of time.
- 5) All control valves to be positioned at grade.
- 6) Preference is for simple and robust facilities and operation.
- 7) Ladder access provided to top of tower.
- 8) Use of instrument impulse lines shall be minimized to decrease pluggage potential.
- 9) The design shall provide for swinging between different feed and product tray locations.
- 10) All fittings shall be accessible.

CONTROL STRATEGY: A suggested control strategy is contained in Section 10 of this document for reference and to give guidance to the design of the control room facilities. This control strategy is considered to be workable, however, other options are available and the final control scheme will be developed in consultation with the package unit vendor.

REUSE OF EXISTING VESSELS

Other than the pressure vessels to be supplied with the package unit, no new pressure vessels or tankage are required by this project. The existing Spent IPA storage tanks will be realigned, one being put into offspec IPA service and one being made available for the Gunk Lugger Replacement project.

7.0 HEAT TRANSFER EQUIPMENT

All exchangers shall be supplied by the package unit vendor. The condenser and reboiler for the distillation tower shall be bayonet type units to minimize equipment needs and simplify operation. The IPA product condenser shall be designed so that the liquid from the exchanger is sufficiently subcooled to eliminate flashing across the downstream control valve.

8.0 PUMPS AND COMPRESSORS

A new rotary positive displacement pump, P1, is required to move the offspec IPA stored in D-538 back to the IPA Refining unit. This pump shall be located at the IPA Storage area as described in Section 4.

A positive displacement pump shall be required to move the bottoms from the IPA tower to the gunk system. This pump will be supplied as part of the package unit.

9.0 MATERIAL HANDLING EQUIPMENT (not required)

10.0 INSTRUMENTATION AND CONTROL

SCOPE

This section contains a description of a proposed control strategy for the package unit so that offsites facilities can be designed correctly and to give a general understanding of the control aspects of the IPA refining column. The final strategy will require discussions with the package unit vendor to finalize.

PROCESS CONTROL STRATEGY

FEED TO IPA COLUMN: Feed to the IPA column will be contained in existing oil tank C-535 where light material will be flashed off. The level in C-535 will be controlled by modifying the feed rate to the IPA column. C-535 has about 3 hours of surge capacity between normal liquid level and high liquid level. The control will be tuned to avoid sharp changes in the feed rate to the IPA column. If the IPA unit is unavailable the feed can be manually swung to the offspec IPA tank, D-538, for later rerunning.

IPA PRODUCT: An analyzer will be provided to measure the purity of the IPA product stream. This will allow modification of the flow rate of the IPA product stream to maximize the draw at a given product purity. The IPA product sidestream will be taken as a vapour off the column to maximize purity and will be condensed in the IPA product condenser. This exchanger shall be arranged so that there is sufficient liquid level even at maximum rates to allow sufficient subcooling of the liquid so that flashing does not occur in the downstream control valve. The analyzer reading will cascade to the level which be maintained by the control valve position.

BOTTOMS STREAM: The flow rate of the bottoms stream will be adjusted to maintain the level in the bottom of the tower. The stream will be combined with the light oil liquid draw from the top tray of the column and pumped to gunk storage at the IPA storage area.

LIGHT OIL DRAW FROM TOP TRAY: To remove the potential azeotrope with water a small liquid stream is taken off the top tray. This stream is flow controlled and is discharged into the bottoms line to be pumped to the gunk storage tank with the bottoms stream.

TOWER PRESSURE CONTROL: An uncondensed vapour stream is taken off the top of the tower to the flare or Vent Gas compressor to control the tower pressure.

OFFSPEC PRODUCTION: When the IPA product goes offspec the analyzer will alarm this condition. If the situation cannot be resolved after a suitable period of time, the IPA product can be manually swung to offspec tank, D-538.

OFFSPEC RERUNNING: When sufficient offspec material has accumulated, an offspec rerunning operation can be initiated. The valving will be manually swung and the offspec pump, P1, started to send offspec material back to the IPA column to be injected with the fresh feed. The normal offspec recycle rate, at 15% of the normal flow rate to the column, is sufficiently low that no problem with the column operation is expected. The provision is made to increase this rate should the situation warrant, by increasing the motor speed.

STEAM FLOW: The steam rate to the tower reboiler shall be controlled to maintain a set tower pressure drop.

CONTROL ROOM EQUIPMENT

Capacity exists within the DCS for the new instrumentation. Termination equipment in the form of slots and cards will be required to input the new signals into DCS, and wiring will be needed to transmit the signals. A summary of the field wiring and control house equipment is as follows:

- 1000 feet of 24 pair shielded cable from IPA package unit to marshalling panel MPHA-1.
- terminalling for the field cables. It is assumed room exists in the marshalling panel terminal blocks.
- two new DCS cables, approximately 50 feet long, one 8 pair and one 16 pair.
- one new analog input card/FTA/cable to be installed in PM #15.
- one new analog output card/FTA/cable to be installed on PM # 15.

PRODUCT ANALYZER

An analyzer shall be installed on the IPA product stream to monitor the IPA product purity and allow maximization of the IPA recovery at a given purity. This analyzer will be located in the existing LDPE analyzer building, where room exists to house it. The sample system will be arranged so that the sample loop returns the sample material back to the process downstream of the product IPA control valve.

The pressure rating of the analyzer components must be suitable to handle the safety relief conditions of the IPA refining tower. This pressure shall be confirmed during the detailed engineering phase of this project.

11.0 SAFETY AND ENVIRONMENTAL

SAFETY VALVES

Several new safety valves shall be provided as part of the package unit. Safety valves will be needed on the top and bottom of the distillation tower for overpressure protection, and on the steam and water piping to the exchangers for overpressure protection. A safety valve will also be needed on the discharge of the bottoms pump.

New safety valves are also specified for the offspec pump discharge and for thermal expansion protection on the offspec return line. The inlet valve to the offspec tank, D-538, shall be car-sealed open to allow a free path for the safety valve discharges. During detailed design consideration shall be given to terminating the safety valve discharge lines between the valve and the tank to eliminate the need for the car-seal.

FIREFIGHTING

An extension to the existing deluge system will be provided to protect the package unit. There is sufficient capacity in the existing system to accommodate the increase in flow required. The location and number of existing fire monitors is sufficient for fire fighting purposes.

ENVIRONMENTAL

Preparation of a Ministry of the Environment Certificate of Approval will be required for this project.

EFFLUENT DISPOSAL

There are no effluent streams as part of this project that reach the atmosphere in a raw state. The overhead vapours from the column are burned in the flare, and the oils and waxes are sent to Laidlaw for environmentally acceptable incineration with the gunk stream.

12.0 PIPING

FLEXIBILITY ANALYSIS

A piping flexibility analysis shall be carried out at the discretion of the package unit vendor for lines contained on the package unit. Because of the largely ambient temperature operation for most of the offsite piping changes, it is not expected that a flexibility analysis will be required for those facilities.

SAFETY VALVE DISCHARGE

The package unit vendor shall ensure that the discharge lines from the safety valves are supported such that the reactive forces during discharge will not result in undue stresses on equipment. Block valves shall be installed on the safety valve discharge piping to allow maintenance of the 3 way valves.

HEAT TRACING

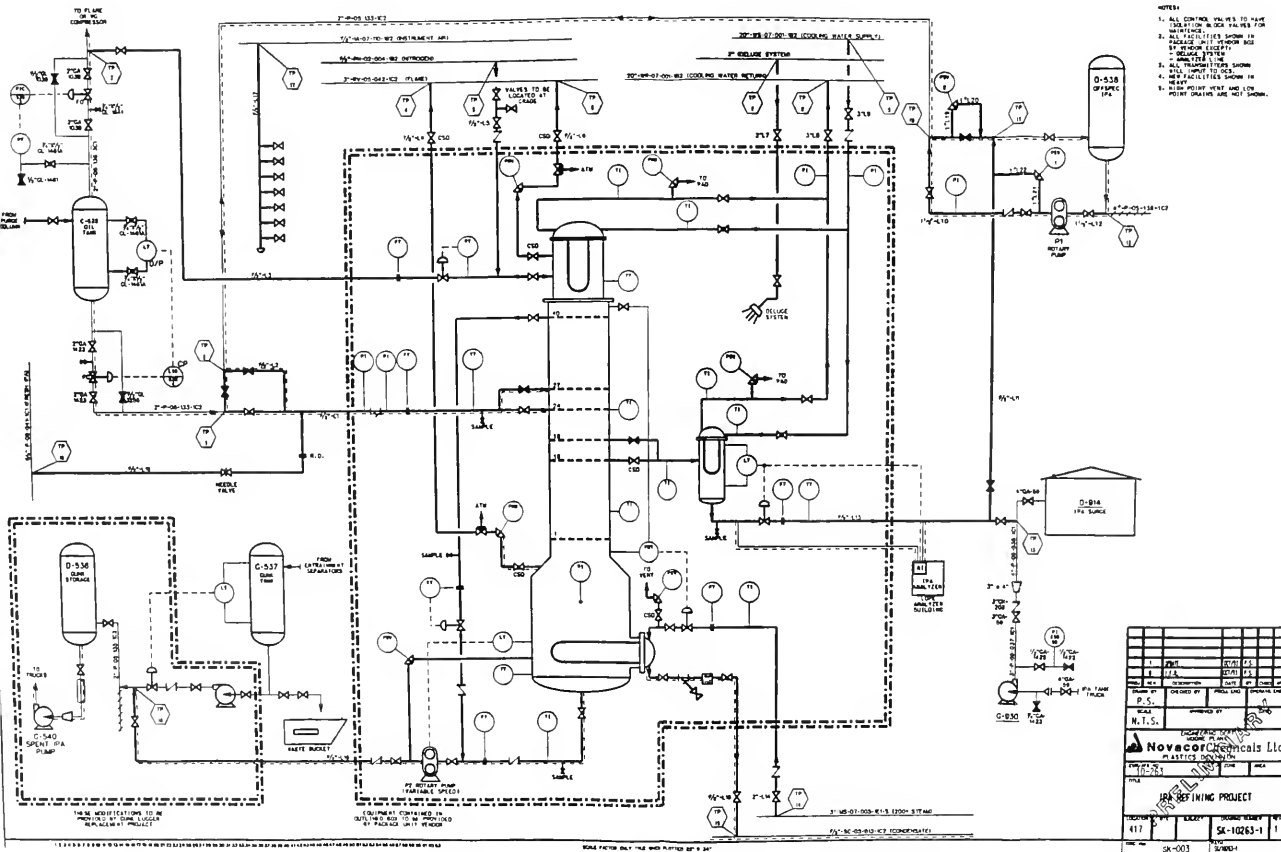
Steam tracing will be needed to prevent setup or freezing on certain lines as detailed on the line schedule. It is expected that 3 new tracers from an existing manifold near the new package unit will satisfy the need in that location. At the IPA storage area it is expected that the existing tracers can be extended to handle the additional piping there.

INSULATION

Insulation shall be installed as detailed on the line schedule with type and thickness in accordance with the Union Carbide standards.

MISCELLANEOUS FITTINGS

A strainer is included as part of the package unit to catch any large chunks of hard waxy material that sometimes enter the Oil Tank from the purge column feed filters. This is an abnormal situation and it is not expected that removal of solids is needed as a part of the normal operation. The strainer mesh size shall be specified to capture 1/2" chunks.



NOTES:

1. ALL CONTROL VALVES TO HAVE ISOLATION BLOCK VALVES FOR MAINTENANCE.
2. ALL FACILITIES SHOWN IN PROCESS UNIT VENDOR BOB SP VENDOR EXCEPT:
- DELUGE SYSTEM
- ANALYZE LINE
3. ALL TRANSMITTERS SHOWN WILL INPUT TO DCS.
4. NEW FACILITIES SHOWN IN HEAVY
5. HIGH POINT VENT AND LOW POINT DRAINS ARE NOT SHOWN

[illegible]

